

Synthesis and structural properties of Mg (OH)₂ on RF sputtered Mg thin films by simple hot water process method

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Abstract - Mg thin films were prepared on commercial glass substrate by RF sputtering and processed in highly pure hot water at 95 °C. Various process time were used to study the growth behavior of Mg (OH)₂ phases. Thin films processed at above 4 hrs duration showed the growth of preferred (001) oriented Mg (OH)₂ phase. The observed XRD spectra also showed the changes on structural properties of processed Mg thin films. The nano (< 12 nm) size crystals were observed in the processed samples. The properties of Mg phase influenced by tensile stress due to Mg (OH)₂ growth. FTIR spectra also evidenced the formation of Mg (OH)₂ over the Mg thin film by hot water process. AFM images revealed the dense nature of hot water processed Mg thin film. Increased particle size was achieved with process time at 1 and 6 hrs process time.

Keywords: Mg thin film; Mg(OH)₂; Hot water process; Xray diffraction; AFM analysis

I. INTRODUCTION

The devices such as mobile phones, laptop computers, cameras, composite materials and other electronic components are prepared by the using material such as Mg since it is lightest among the metallic one. Now a days, Mg has been considered as an alternative for Al because of its attractive properties such as 35% weightless than Al, good mechanical, electrical properties and good vibration resistivity. But it has poor corrosion resistivity in all environments. For new application in various fields, a new surface coating would be used to improve its corrosion resistivity. Moreover, the structural and other properties of thin film material are different from that of the bulk material [1]. In addition to their applications as elemental form, it combines with other

material and showed good performance e.g. Mg₂Si, has attracted much interest as a narrowgap semiconductor and environmentally friendly material [2], and thermoelectric power devices [3]. Because of its excellent mechanical properties and high corrosion-resistance, Mg₂Si was used and applied to structural components [4].

In addition to the above, Mg based switchable mirror are also attractive materials for regulation of light and heat transfer in buildings, vehicles, satellites and many more applications in sensor technologies. It is necessary to understand the influence of environmental condition on the properties of Mg based thin film for the specific applications. A window using this technology can be programmed to respond to local sunlight and weather conditions, optimizing the amount of daylight entering a building, while controlling the gain or loss of infrared radiation or heat [5]. Recently, Richardson et al. [6] reported the influence of 3d transition metals such as Ni, Mn, Fe, and Co on the properties of Mg thin films. Magnesium can be deposited as thin film by means of various techniques under vacuum conditions.

Many research papers have been published based on the preparation of Mg thin film by physical vapor deposition [7-9]. Recently, Y. K. Gautam et al. have prepared Mg thin film by DC sputtering and reported their properties [10]. Still there are no reports published based on the reaction at the surface of the Mg thin film and their products. Magnesium hydroxide (Mg(OH)₂) is one among the derivatives of Mg thin film and has some attractive properties such as fire retarding, halogen-free flame retardant filler in composites materials, catalysis, and additive in refractory, paint, and ceramics brings [11]. When it undergoes endothermic decomposition at 332 °C, it produces MgO and water as byproduct.



The heat absorbed by the reaction acts as a retardant by delaying ignition of the associated substance. The

resultant product, water released during the reaction dilutes any combustible gases and inhibits oxygen from aiding the combustion [12]. A simple, mild, and practical method for the low-cost and large-area fabrication of high-quality $\text{Mg}(\text{OH})_2$ is still a challenge for material scientists and chemists. In this paper, the rf sputtered Mg thin films were processed in high pure hot water at various time durations and their structural and surface properties have been reported here. It is also an attempt to synthesize of $\text{Mg}(\text{OH})_2$ over Mg thin film for fire retardant applications.

II. EXPERIMENTAL METHODS

Mg thin films were deposited on glass substrates using Mg (99.99% purity) target (3 inch in diameter and 5 mm in thickness) by RF sputtering (Edwards make, Model-Auto 500). The base pressure of the chamber was $\sim 2 \times 10^{-6}$ torr. High pure (99.999%) Ar was used in sputtering. The sputtering pressure was 3.0×10^{-3} mbar during the synthesis of Mg thin films. The substrates were cleaned by rinsing in ultrasonic bath of acetone and isopropyl alcohol. To get 1.5 μm film thicknesses, the deposition time and sputtering power were kept fixed at 45 min and 300 W, respectively. All Mg thin films were coated at room temperature. The target was pre-sputtered for 5 min before starting deposition. To get the uniform thickness, rotary drive system was used and 25 rpm was fixed for all Mg film coatings. Substrate to target distance of 15 cm was kept as constant for all depositions.

Ultra high pure hot water (resistivity more than $18.2 \text{ M}\Omega \text{ cm}$) treatment was performed in 500 ml beaker at 95°C , using a temperature controlled magnetic stirrer for the specimen coated with metallic Mg thin film. All Mg thin film samples are dipped in ultra high pure hot water for five different times (1 hr, 2 hrs, 4 hrs, 5 hrs and 6 hrs). To verify the hot water treatment, all prepared films are treated with ultra high pure water at room temperature for the said time durations. The crystalline nature of the as-grown and processed Mg thin films was investigated by using a high resolution X-ray diffraction (HRXRD, X'pert-PRO, Philips, Netherlands). A $\text{CuK}\alpha$ ($k = 1.54056 \text{ \AA}$) source was used, with a scanning range between $2\theta = 20^\circ$ and 80° . The surface properties of the as grown and processed films were analyzed by using AFM system (Dimension Edge – Bruker AXS make). The FTIR spectra were recorded in the range $400\text{--}4000 \text{ cm}^{-1}$, with a spectral resolution of 4 cm^{-1} using Perkin Elmer spectrum GX.

III. RESULTS AND DISCUSSION

A. Structural Properties

The XRD spectrum of as grown and hot water processed Mg thin films was recorded as shown in Fig.1. No reaction can be observed for all films treated in ultra high pure at room temperature. Since the results of the film processed at 3 hrs duration shows almost similar and no difference could be observed from the sample processed at 2 hrs duration, the results of the samples treated at 3 hrs duration are not discussed here. It depicts that the as grown and processed samples are polycrystalline nature with an intensive peak at $2\theta \sim 34.25^\circ$ corresponding to a hexagonal closed packed (hcp) structure reflected from (002) orientation. The reason for this highly intense (002) orientation of Mg is the low energy configuration corresponding to this plane [13]. The observed peaks were indexed as the hexagonal structure and compared with the standard data of both Mg (JCPDS Card No: 897195) and $\text{Mg}(\text{OH})_2$ (JCPDS Card No:441482).

The observed peak positions and structural parameters are given in Table 1 and 2. No oxide or hydroxide peaks are observed with as grown films. In addition, two more peaks related to cubic (200) and hexagonal (004) phase are also observed with very low intensity in the as grown film. But the peak related to (002) plane was observed at lower 2θ when compared to standard 2θ position (JCPDS No. 897195). This may be due to stress developed during deposition process. Substrate (SiO_2) related peaks were also observed at $\sim 2\theta = 30.80^\circ$ in all samples. The preferred (002) plane could be possible only with high Ar pressure during the sputtering process which exhibits granular and fine structure [14].

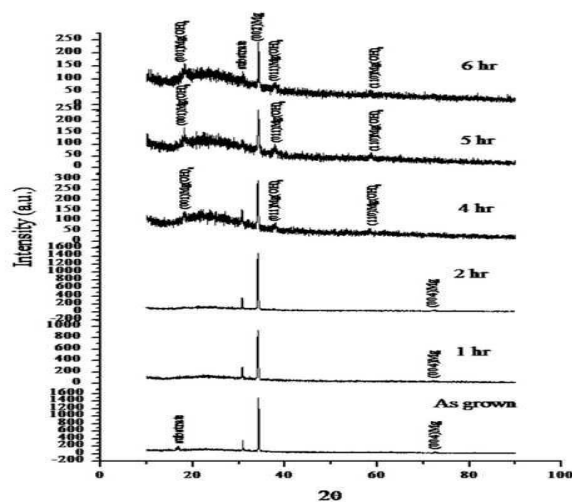


Figure 1 XRD spectra of as grown and hot water processed Mg thin film for various time

TABLE I THE STRUCTURAL PROPERTIES OF AS GROWN AND HOT WATER PROCESSED Mg THIN FILM

Process time	Phase	2 θ	FWHM ($^{\circ}$ 2 θ)	d (Å)	Crystallite size (nm)	Internal stress (GPa)	Micro Strain (ϵ)	Dislocation density δ ($\times 10^{20}$ lin/m 2)	JCPDS No.
As grown	H(002)	34.29	0.144	2.613	10.52	-0.0198	3.44	0.904	897195
	C(200)	63.11	---	1.472	---	0.0207	---	---	657219
	H(004)	72.54	0.192	1.304	9.36	-0.0075	3.87	1.41	897195
1 hr	H(002)	34.25	0.207	2.616	7.32	-0.0273	4.95	1.87	897195
	H(004)	72.44	---	1.303	---	-0.0015	---	---	897195
2 hrs	H(002)	34.28	0.185	2.614	8.19	-0.0223	4.42	1.49	897195
	H(004)	72.38	---	1.304	---	-0.0060	---	---	897195
4 hrs	H(002)	34.22	0.244	2.618	6.21	-0.0323	5.83	2.59	897195
5 hrs	H(002)	34.23	0.243	2.618	6.24	-0.0323	5.81	2.57	897195
6 hrs	H(002)	34.29	0.199	2.613	7.62	-0.0198	4.75	1.72	897195

TABLE II THE STRUCTURAL PROPERTIES OF Mg(OH) $_2$ PHASE OBSERVED FROM THE HOT WATER PROCESSED Mg THIN FILM

Process time	Phase	2 θ	FWHM ($^{\circ}$ 2 θ)	d (Å)	Texture coefficient	Crystallite size (nm)	Internal stress (GPa)	Micro Strain ϵ ($\times 10^{-2}$ lin $^{-2}$ m $^{-4}$)	Dislocation density δ ($\times 10^{20}$ lin/m 2)	JCPDS No.
4 hrs	H(001)	18.18	0.324	4.877	1.19	4.53	-0.1226	8.00	4.87	441482
	H(011)	37.79	---	2.379	0.44	---	-0.03031	---	---	822453
	H(110)	58.51	---	1.576	1.37	---	-0.00414	---	---	822453
5 hrs	H(001)	18.21	0.042	4.869	2.15	34.92	-0.10898	1.04	0.082	441482
	H(011)	37.85	0.468	2.375	0.40	3.27	-0.01929	11.06	9.35	822453
	H(110)	58.74	---	1.566	0.45	---	0.037286	---	---	822453
6 hrs	H(001)	18.24	0.368	4.859	1.31	3.98	-0.09535	9.08	6.4	441482
	H(011)	37.84	0.282	2.376	0.33	5.43	-0.02204	6.67	3.39	822453
	H(110)	58.55	---	1.575	1.36	---	-0.01243	---	---	822453

In addition to (002) Mg peak, it is also noticed that the XRD spectra of films processed at above 4 hrs duration show the presence of some Mg(OH) $_2$ peaks with different orientations. The spectra also show the presence of hexagonal related (001), (011) and (110) peaks of Mg(OH) $_2$ when the process time increases at and above 4 hrs. In order to study the structural properties in detail, the structural parameters are calculated as given in Table 1 and 2. Since the peak position and intensity seem to be important factors which decide the crystalline quality, the intensity analysis has also been considered in this study. Fig. 2 shows the change in intensity of (002) peak of Mg for various process time. It depicts that the Mg film processed at 2 hrs shows a higher value in intensity than the film processed at various time duration that has the improved crystalline quality of such films. It is also noticed that the peak width increases as the process time increases. It also reveals that the films processed at above 4 hrs duration shows low intensity peaks compared to 1 hr and 2 hrs durations. It is attributed to the growth of Mg(OH) $_2$ from Mg for long process time. Fig. 2 also shows the change in peak position towards left when the

process time increases from 1 hr to 5 hrs. Thereafter, the peak position does not change as observed with as grown Mg thin film. Fig. 3 shows the peak of (004) plane with respect to process time. It is visible that the (004) peak diminishes when the film processed at and above 4hrs duration in hot water. It is also noticed that the position of the peak slightly moves towards left as observed for (002) peak. Fig. 4 (a-c) shows the change in intensity of Mg(OH) $_2$ related peaks with process time. Fig. 4(a) clearly indicates that the intensity of (001) oriented peak increases with process time upto 5 hrs and reduces at 6 hrs. The film processed at 6 hrs duration shows very broad peak which seems to be the nature of low crystallite size. Fig. 4(b) represents the variation of intensity of (011) peak and shows that the film processed at 5 hrs duration exhibit the higher intensity as well as broad. It also reveals that all peaks are broadened and seems to be the peak merging. Fig. 4c shows the change in intensity of (110) peak and also shows the higher intensity for the film processed at 5 hrs duration. It also shows the growth behavior of (011) and (110) phases at this process time.

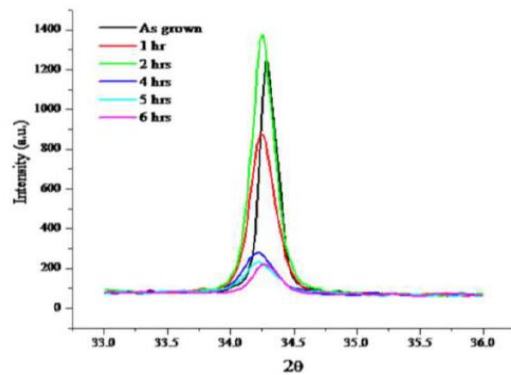


Figure 2 Variation in peak intensity and position of the (002) plane of Mg phase

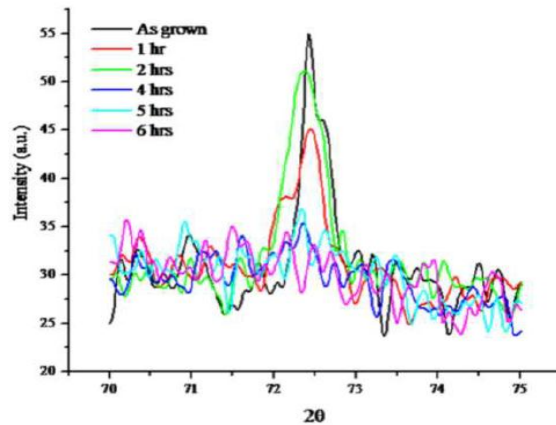


Figure 3 Variation in peak intensity and position of (004) plane of Mg phase

A considerable reduction in intensity could also be observed for the process time longer than 5 hrs. Moreover a small shift in peak position for all (001), (011), and (110) peaks towards right angle could also be observed with peak broadening.

In order to understand the influence of hot water process in structural properties of Mg thin film, a detailed structural analysis is required and hence the structural parameters of the processed film were calculated from the observed XRD spectra. The calculated structural parameters are given in Table 1. Table 2 shows the structural parameters of Mg (OH)₂ phase formed on the surface of the processed samples. In order to investigate the possibility of preferred orientation, the Harris analysis [15] was performed using the following relationship for the texture coefficient.

$$P_i (TC) = N (I_i/I_0) / \sum N_i = 1(I_i/I_0) \quad (1)$$

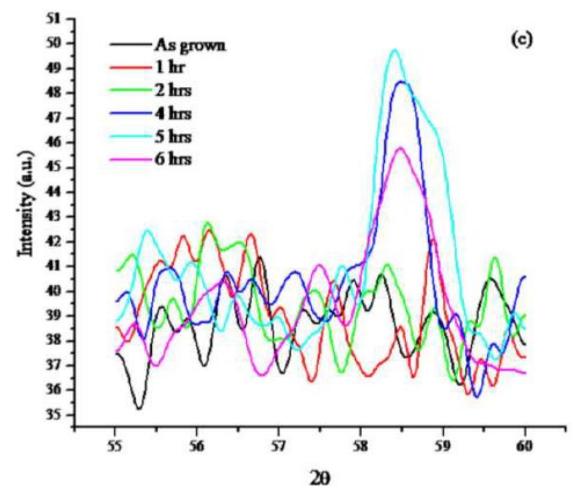
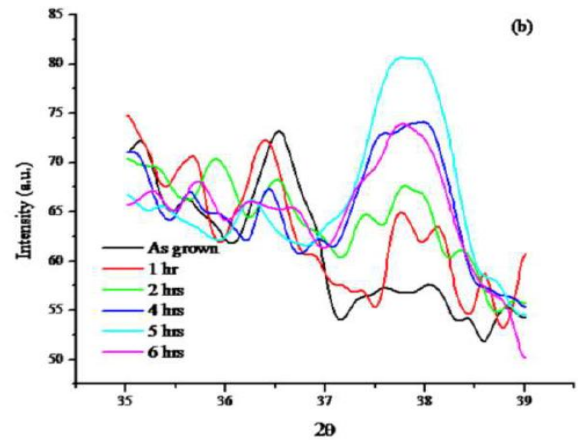
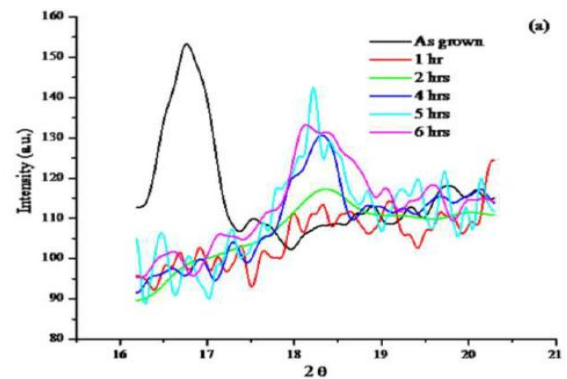


Figure 4 Change in intensity of Mg(OH)₂ related peaks reflected from (a) (0 0 1), (b) (0 1 1), and (c) (1 1 0) planes

Where P_i is the Texture Coefficient of the plane i , I_i is the measured intensity, I_0 is the intensity of the JCPDS powder diffraction pattern of the corresponding peak and N is the number of reflections considered for the analysis. P_i is unity for each reflection in the case of a randomly oriented sample and values of P_i greater than unity indicate preferred orientation of the crystallites in that particular direction. Fig. 5 shows the variation of texture coefficient of $\text{Mg}(\text{OH})_2$ related phases. It exhibits that the (001) peak shows high value for the film processed at 5 hrs duration and afterwards it decreases considerably for longer process time (>5 hrs). A reverse behavior could also be observed for (110) phase. It shows that 5 hr process time enhance the growth of (001) plane of $\text{Mg}(\text{OH})_2$ phase and hence the preferred (001) oriented growth is achieved. Overall, the Mg films processed at 4 hrs and 6 hrs in hot water helps to grow the preferred oriented $\text{Mg}(\text{OH})_2$ phase with (001) and (110) planes. It is also noticed that the (011) oriented $\text{Mg}(\text{OH})_2$ phase grows randomly over on Mg thin film when processed in hot water at more than 4 hrs duration.

The crystallite size (D) was calculated using the Debye Scherer formula [16] from the Full-Width at Half-Maximum (w) measurements:

$$D = 0.94\lambda / w \cos\theta \quad (2)$$

Where, λ is the wavelength of incident X-ray. The calculated crystallite sizes of all films are given in Table 1 and 2. The change in crystallite size of (002) Mg plane is graphically represented in Fig. 6. It reveals that the crystallite size decreases gradually as the process time increases. It seems that the crystallite size of as grown and films processed at 4 and 5 hrs durations shows high value and low value respectively. Afterwards, an increasing manner in crystallite size could also be noticed with the films processed at longer than 5 hrs durations. Based on the peaks observed from $\text{Mg}(\text{OH})_2$ phases, the crystallite size could be evaluated for (001) and (011) peaks only (see Table 2). It depicts that the crystals size of (001) plane show high value (34.92 nm) when the film processed for 5 hrs duration. For (011) plane, a small increase in crystallite size from 3.27 nm to 5.43 nm is observed as the process time increases from 5 hr to 6 hrs duration.

The internal stress (σ) in the processed samples is calculated using the relation

$$\sigma = -E (da - do) / (2doY) \quad (3)$$

where do and da are the d spacing of bulk Mg and thin film forms respectively. E and Y are the Young's modulus and Poisson's ratio of Mg respectively. The Young's modulus and Poisson's ratio of Mg is $E = 45$ Gpa and $Y = 0.29$ respectively [17]. From Table 1, it is observed that the calculated stress from the XRD results shows negative sign. It seems to be the behavior of applied stress for (002) orientation during the process time as tensile stress.

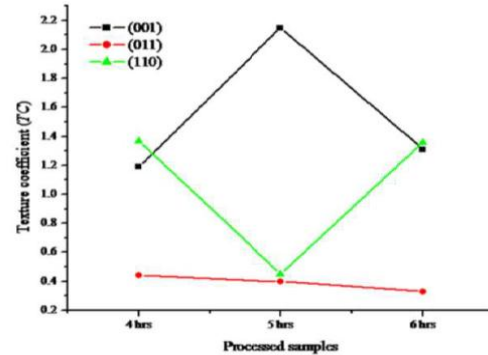


Figure 5 Calculated texture coefficient of $\text{Mg}(\text{OH})_2$ planes for various process time.

It also depicts that the change in internal stress is observed as increasing for the film processed upto 5 hrs duration. It is also noticed that the high value in micro strain of (002) plane could be observed with the thin film processed at 4 hrs duration. Overall the hot water process plays an important role to increase the micro strain value of (002) plane.

To strengthen this point, the dislocation density (δ), defined as the length of dislocation lines per unit volume of the crystal, was evaluated from the formula [18];

$$\delta = 1 / D^2 \quad (4)$$

It is a term to define the crystal quality with defects. Overall, the calculated dislocation density is found to increase for hot water process. It shows that the high values of $2.59 \times 10^{20} \text{ lin/m}^2$ and $2.57 \times 10^{20} \text{ lin/m}^2$ are observed for the films processed at 4 hrs and 5 hrs duration respectively. Afterwards, the dislocation density decreases as process time increases. From the formula, it is clear that the crystallite size plays a vital role in dislocation density. In addition, the (001) plane of $\text{Mg}(\text{OH})_2$ phase has also influence on the crystal quality of Mg (002) plane when Mg thin film processed at 4 hrs duration. The results show that it can also be applicable for the observation made for the strain analysis too.

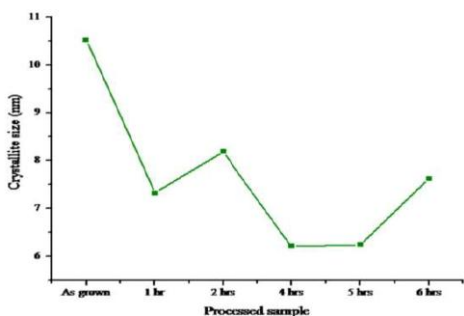


Figure 6 Change in crystallite size of the hot water processed Mg thin films for various process time

In order to analyze the structural properties of $\text{Mg}(\text{OH})_2$ phase, the structural parameters are calculated from the XRD spectra for the samples processed at above 4 hrs duration as given in Table 2. In this $\text{Mg}(\text{OH})_2$ phase, the applied stress during the growth is tensile nature. It is observed that the applied stress for both (001) plane and (011) plane decreases gradually as the process time increases from 4 hrs to 6 hrs. But a change from tensile to compressive could be observed with the (110) plane when the process time increases from 4 hrs to 5 hrs. Afterwards, the conversion from compressive to tensile is observed when process time increases upto 6 hrs. From the Table 2, it is observed that the similar behavior on dislocation density and microstrain could also be observed for the $\text{Mg}(\text{OH})_2$ phase. In detail, high value in microstrain (11.06) and dislocation density ($9.35 \times 10^{10} \text{ lin/m}^2$) of (011) plane is observed for the film processed at 5 hrs duration. A noticeable reduction on both microstrain and dislocation density is also observed for (001) plane when the process time increases from 4 hrs to 5 hrs. Afterwards, the values are increasing for the film processed at 6 hrs durations. For (011) plane, the values are decreasing noticeably for increasing the process time from 5 hrs to 6 hrs.

C. FTIR analysis

The observed IR spectra of as grown and hot water processed Mg thin films are shown in Fig. 7a. As grown Mg thin films show no peaks related to their compounds. The evidence of the presence of $\text{Mg}(\text{OH})_2$ was observed in FTIR absorbance spectrum for all samples processed at above 4 hrs in hot water, as shown in Fig. 7(a). A sharp and intense absorption peaks observed at 3699 cm^{-1} assigned as vibrational frequency of $-\text{OH}$ from the magnesium hydroxide compounds [19]. It seems to be the evidence of the formation of hexagonal $\text{Mg}(\text{OH})_2$ on the surface of Mg thin film by hot water process. But these

peaks are only observed with the films processed at above 4 hrs durations. It is evidently proved by XRD results. The peaks observed for the films processed at 1hr and 2 hrs durations may be related to the stretching vibration of $-\text{OH}$ from water molecules adsorbed on the Mg thin film surface. The intensity of the film processed at 2 hrs duration shows higher value than 1 hr process time, which may be due to the adsorption of more water molecules on the film surface.

The process time plays an important role on not only changing the intensity of peak and also the peak position observed at around 3699 cm^{-1} . The change in intensity and position of the peak observed at $\sim 3699 \text{ cm}^{-1}$ is given in Fig. 7b. It shows that the stretching vibration peak is observed between 3699 and 3703 cm^{-1} for the film processed at above 4 hrs duration.

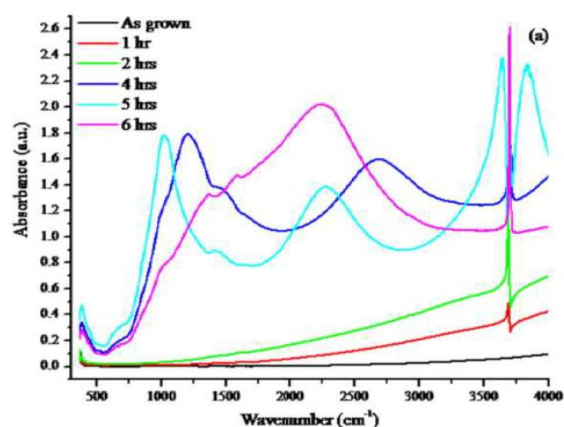
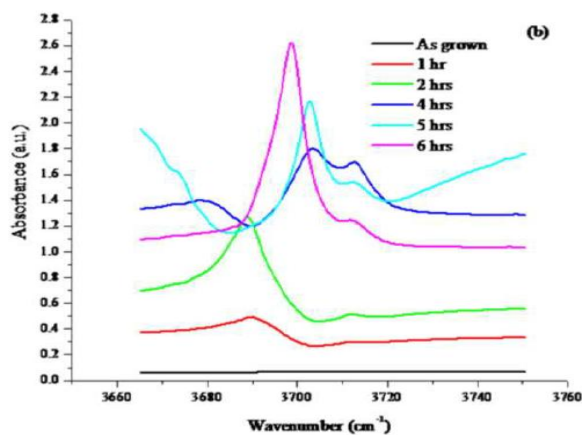


Figure 7 (a) FTIR spectra of hot water processed Mg thin films for various time duration and (b) IR spectra recorded in between 3665 and 3750 cm^{-1} for various process time durations.



The Mg thin films processed at 5 hrs and 6 hrs show intense peak at 3703 and 3699 cm^{-1} with broad shoulder peak at around 3712 cm^{-1} respectively. This may be attributed to the isolated surface OH groups of the processed films [20]. The Fig. 7(b) also shows that the film processed at 4 hrs duration shows a broad peak at 3703 cm^{-1} with shoulder peak (3712 cm^{-1}). It is also noticed that the intensity of shoulder peaks diminishes as the intensity of main peak increases, which indirectly seems to be the influence of increase in hot water process

duration. Overall, the intensity get increases as the process time increases and high intense peaks are observed with the Mg films processed at above 6 hrs process time. The peak position at higher frequency is observed with only 4 and 5 hrs processed samples. The peaks (3430 cm^{-1}) related to the transformation from free protons into a proton-conductive state in brucite could not be observed for all process conditions.

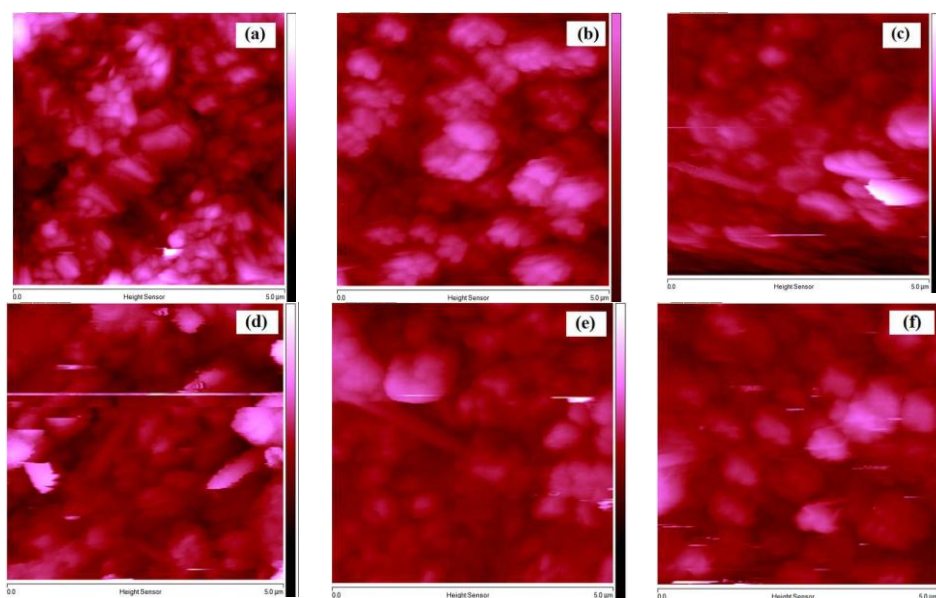


Figure 8 AFM surface morphology of (a) as grown, (b) 1 hr, (c) 2 hr, (d) 4 hrs, (e) 5 hrs, and (f) 6 hrs hot water processed Mg thin films

A strong peak observed at around 3636 cm^{-1} and 3830 cm^{-1} can be assigned to hydrogen-bonded hydroxyl groups and $\text{Mg}(\text{OH})_2$ [21]. It is observed only with the film processed at 6 hrs duration. In addition to this, a small absorption peak at 1406 cm^{-1} was attributed to the $-\text{OH}$ bond bending vibration in crystal structure [22]. Few unidentified peaks at $\sim 1020\text{ cm}^{-1}$, 2282 cm^{-1} , 2250 cm^{-1} and 2680 cm^{-1} could also be observed. This may be due to the impurities on the surface of the film.

D. Surface analysis

TABLE 3 SURFACE ROUGHNESS AND PARTICLE SIZE OF

Sample name	Roughness (nm)	Particle size (nm)
As grown	68	250
1 hr	59	616
2 hrs	123	345
4 hrs	90	249
5 hrs	91	166
6 hrs	62	386

In order to study the surface properties, the surface morphology of the films was recorded by AFM as given in Fig. 8. Fig. 8(a) shows the surface morphology of as grown Mg thin film. From the Fig. 8 (b-f), it depicts that the noticeable change on grain growth as well as the surface morphology of the films are observed for the hot water processed film.

It clearly indicates that the hot water processed samples is more dense than the as grown. Particles agglomeration could be easily viewed for the samples processed at 1 hr duration. In addition, the surface morphology of the films processed at 4 hrs and 5 hrs shows similar properties and uniform surface could also be achieved with the film processed at 6 hrs duration. The particle size and surface roughness of the as grown and hot water processed thin films are calculated directly from the AFM image and given in Table 3.

It shows that the roughness increases drastically for the sample processed at 2 hrs duration. It is also noticed

that the low value in surface roughness could be observed for 1 hr and 6 hrs processed samples. In particle size analysis, the table shows high value (616 nm) for the film processed at 1 hr duration and low value (166 nm) for the film processed at 5 hrs duration. Overall the particle size suddenly increases from 250 to 616 nm and gradually decreases from 616 to 166 nm as changing the process time from 1 hr to 5 hrs durations.

V. CONCLUSION

RF sputtered Mg thin films were processed with high pure hot water at 95°C for different time durations. The XRD spectra revealed the presence of (002) oriented Mg peaks in all samples and also depicted the growth of Mg (OH)₂ phases in all samples processed at and above 4 hrs duration. Structural parameters showed the influence of Mg (OH)₂ on the structural properties of Mg phase in processed samples. The tensile stress developed by Mg (OH)₂ phase influenced the stress of Mg phase. The observed crystallite sizes of both Mg and Mg (OH)₂ phases were found within 12 nm except with (001) plane of Mg (OH)₂ for 5 hrs process time. Preferred growth in Mg (OH)₂ phases was achieved for (001) plane in the films processed at > 4 hrs duration. FTIR spectra showed an evidence for the formation of Mg (OH)₂ on the surface of the hot water processed films. AFM images revealed the improved film density for the film processed at and above 2 hrs durations. Noticeable changes in surface roughness and particle size for the hot water processed samples were achieved. Based on these results, it is suggested that the growth of preferred Mg (OH)₂ phases is possible over Mg thin film by hot water process method and may be used for industrial application where the fire retardant is needed.

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